

# TiNi-based thin films for MEMS applications

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**Abstract**—In this paper, some critical issues and problems in the development of TiNi thin films were discussed, including preparation and characterization considerations, residual stress and adhesion, frequency improvement, fatigue and stability, as well as functionally graded or composite thin film design. Different types of MEMS applications were reviewed and the prospects for future advances in fabrication process and device development were discussed.

**Index Terms**—Shape-memory; TiNi; Sputtering; Thin Films; MEMS, Microactuator; Microsensor

## I. INTRODUCTION

Shape memory alloys (SMAs) possess an array of desirable properties: high power to weight ratio, thus the ability to recover large transformation stress and strain upon heating and cooling, pseudoelasticity (or superelasticity), high damping capacity, good chemical resistance and biocompatibility. This attracted much attention to the research of SMAs as smart (or intelligent) and functional materials [1-3]. More recently, thin film SMA has been recognized as a promising and high performance material in the field of micro-electro-mechanical system (MEMS) applications, since it can be patterned with standard lithography techniques and fabricated in batch process [4-8]. Thin film SMA has only a small amount of thermal mass to heat or cool, thus the cycle (response) time can be reduced substantially and the speed of operation may be increased significantly. The work output per volume of thin film SMA exceeds that of other micro-actuation mechanisms. The phase transformation in SMA thin film is accompanied by significant changes in the mechanical, physical, chemical, electrical and optical properties, such as yield stress, elastic modulus, hardness, damping, shape recovery, electrical resistivity, thermal conductivity, thermal expansion coefficient, surface roughness, vapor permeability and dielectric constant, etc. These changes can be fully made use of the design and fabrication of microsensors and microactuators. However, due to the lack of full understanding of the thin film SMAs together with the difficulty in controlling of the deposition parameters, they have not received as much attention in the MEMS technology as other microactuator technologies.

In this paper, recent advances and development for TiNi SMA thin films were reviewed. Different types of MEMS applications and the prospects for future advances in fabrication process and device development of TiNi films were discussed.

## II. MEMS REQUIREMENTS FOR TINI FILMS

Successful implementation of TiNi micro-actuators requires a good understanding of the relationship among processing, microstructure and properties of TiNi films. The enabling technologies for TiNi films required include: (1). Low-cost, reliable and MEMS-compatible deposition methods with precise control of film composition and quality; (2). Reliable and precise characterization technologies for various properties (such as shape memory effect, superelasticity and mechanical properties, etc.); (3). An appropriate post-deposition annealing (for film crystallization) or aging process compatible with MEMS process; (4). Precise etching and patterning of TiNi film compatible with MEMS process and the possibility of nano-size TiNi structures and actuators; (5) Prediction and modeling of non-linear behavior of TiNi films as well as design and simulation of TiNi thin film microactuators. Some basic requirements for TiNi films used in MEMS applications are listed as follows: (1) Low residual stress to prevent deformation of MEMS structure; (2) High actuation speed and fast response with precise control of deformation and strain; (3) Good adhesion on substrate (free of cracking, delamination and spallation); (4). Durable and reliable shape memory effects; (5) Wide range choice of working temperatures (from below zero to several hundred degree C); (6) Good resistance to surface wear and corrosion; (7) Biocompatible (in case of application in bio-MEMS)

## III. TINI THIN FILM PROCESSING AND CHARACTERIZATION

### A. Sputtering deposition of high quality TiNi films

TiNi based films are the most frequently used thin film SMA materials and they are typically prepared using sputtering method. Laser ablation, ion beam deposition, arc plasma ion plating, plasma spray and flash evaporation were also reported but with some intrinsic problems, such as non-uniformity in film thickness and composition, low deposition rate, or non-batch processing, incompatibility with MEMS process, etc. Transformation temperatures, shape memory behaviors and superelasticity of the sputtered TiNi films are sensitive to metallurgical factors (alloy composition, contamination, thermo-mechanical treatment, annealing and aging process, etc.), sputtering conditions (co-sputtering with multi-targets, target power, gas pressure, target-to-substrate distance, deposition temperature, substrate bias, etc.), and the application conditions (loading

conditions, ambient temperature and environment, heat dissipation, heating/cooling rate, strain rate, etc.).

Precise control of Ti/Ni ratio in TiNi films is of essential importance. The intrinsic problems include the difference in sputtering yields of titanium and nickel at a given sputtering power density, geometrical composition uniformity over substrate and along cross-section thickness of the coating, as well as erosion and roughening of targets during sputtering. To combat these problems, co-sputtering of TiNi target with another Ti target, or using two separate single element (Ti and Ni) targets, or adding titanium plates on TiNi target are widely used. Substrate rotation, good configuration of target position and precise control of sputtering conditions, etc. are also helpful. Varying the target temperature can also produce the compositional modification: sputtering the heated TiNi target can limit the loss of Ti, thus improving the uniformity of film properties. In order to deposit films without columnar structure (thus with good mechanical properties), a low processing pressure of Ar gas (0.5 to 5 mTorr) is essential. Application of bias voltage during sputtering could modify the film microstructure, texture and stress, thus is also important, but few studies have been reported on this topic so far.

TiNi films sputtered at room temperature are usually amorphous, thus post-sputtering annealing is a must because SMA effect only occurs in materials of crystalline form. However, Martensite transformation and superelasticity of TiNi films are sensitive to post-annealing and/or aging temperature and duration. Long term post annealing and aging process should be avoided since it could trigger dramatic changes in film microstructure (i.e., precipitation), mechanical properties and shape memory effects. Films deposited at a relatively high temperature (about 400°C) is crystallized in-situ, thus there is no need for post-annealing. Recently a localized laser annealing method was studied for TiNi films in our group [9], where only certain areas of the film are annealed by laser beam to exhibit shape memory effect (see Fig. 1), and the other non-annealed areas remain amorphous, thus acting as a pullback spring during cooling process. This method opens a new way for fabrication of microdevices.

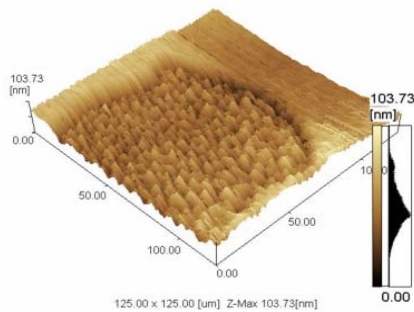


Fig. 1. AFM surface morphology of TiNi films treated with laser annealing

### B. Characterization of TiNi films

For freestanding TiNi films, conventional methods, such as differential scanning calorimetry (DSC), tensile tests

(stress-strain curves) and X-ray diffraction (XRD) are quite applicable. The stress-strain responses of freestanding films are commonly evaluated using tensile tests. For MEMS applications, the TiNi films are usually deposited on Si or related other substrates. One of the important issues in characterization of the TiNi films for MEMS applications is how to correctly evaluate the shape memory effects and mechanical properties of the constrained thin films on substrates. For this purpose, curvature and electrical resistivity measurements (ER) are widely used. Some new methods based on MEMS testing, such as bulge testing, TiNi/Si diaphragm, cantilever bending or damping are more appropriate for microactuator applications, which are compatible with small dimensions and high sensitivities.

In our group, an AFM based in-situ testing method have recently been applied to characterize the phase transformation behavior of the constrained films. Fig. 2 shows two micrographs of AFM surface morphology of TiNiCu films at a low temperature (martensite) and a high temperature (austenite), respectively. The surface roughness of the martensite phase is much higher than that of the austenite. With the change of temperature, the surface roughness values change drastically when transforming between the martensite and the austenite phases, thus clearly reveal the occurrence of phase transformation. The optical reflection changes caused by the changes in the surface roughness and reflective index can also be used to characterize the transformation behaviors of TiNi films. The advantages of this method are its nondestructive nature and applicability to very small size films (down to nanometers).

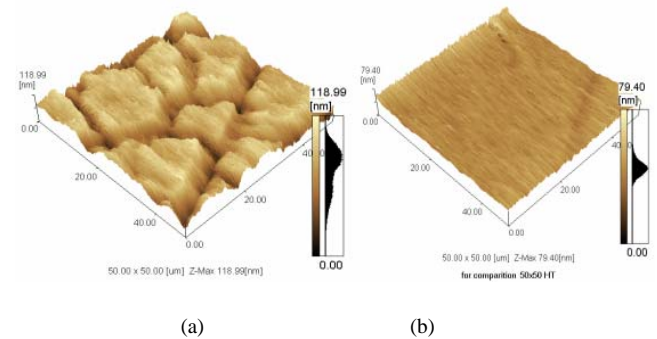


Fig. 2. AFM surface morphologies of TiNiCu films (a) low temperature in martensite state and (b) high temperature in austenite state

In film characterization, there are still many important issues unresolved: (1) Nucleation and growth mechanisms of TiNi thin films and substrate effects; (2) Effects of precipitation, point defects and dislocations; (3) Grain size effect, nano-grain and nanocrystalline structure on shape memory effect and phase transformation. The refinement of grain size can strongly modify the structural and thermodynamic properties, thus the mechanical properties of shape memory alloys; (4) Film thickness effect (since a minimum thickness is required for shape memory effect); (5) Formation of film texture and its control, and the effects on shape memory effect; (6) Internal and external stress on the arrangement of martensite variants, stress induced

martensite and its shape memory phenomenon, etc. (7) Surface chemistry, surface adsorption and biocompatibility of TiNi films with small grain size.

### C. Residual stress and stress evolution

Residual stress and stress evolution in the films could pose potential problems in applications, as it may influence not only adhesion between film and substrate, but also deformation of MEMS structure, mechanics and thermodynamics of transformation and superelasticity effects, etc. Large residual stress could lead to either film cracking or decohesion under tension, or film delamination and buckling under compression. A wide range of residual stress levels (either tensile or compressive) were found in the sputtered films. Deposition conditions, post-deposition thermo-mechanical treatment and composition in TiNi films could have important consequences with respect to the development of residual stress [11,12]. For a film-substrate system, possible origins of stress in thin films can be divided into three groups, i.e., thermal stress, intrinsic stress and phase transformation stress. In crystalline TiNi films, large tensile stress is generated during heating due to the phase transformation from martensite to austenite, while during cooling, the martensitic transformation occurs and the tensile stress drops significantly from the formation and alignment of twins and shear-variant boundary motion, etc. The stress generation and relaxation behaviors upon phase transformation are significantly affected by film composition, deposition and/or annealing temperatures, which strongly control the formation and evolution of intrinsic stress, thermal stress and phase transformation behaviors [13]. An example is shown in Fig. 3. The difference in residual stress for films with different Ti contents can be attributed to the differences in phase transformation behavior, intrinsic stress in the films, and/or precipitates in the films.

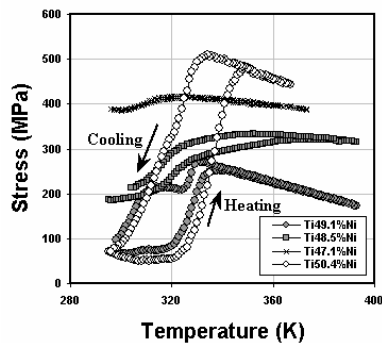


Fig. 3. Stress evolution of TiNi films with different Ti/Ni ratios deposited at a temperature of 723 K

Substrate effect is also significant in the stress generation and evolution, because the difference in thermal expansion coefficients between substrate and TiNi films significantly affects the thermal stress. So far, most studies have been

focused on Si based substrates for MEMS applications. TiNi deposited on other substrates (with different coefficient of thermal expansion) could have different stress state (compressive or tensile) and stress-temperature evolution behaviors, thus detailed studies of substrate effect and stress state on shape memory effect, phase transformation and mechanical properties of TiNi films deserve more and systematic effort [14].

In order to minimize the residual stress in TiNi films, it is necessary to: (1) precisely control the Ti/Ni ratio; (2) deposit films at a possible lower pressure; (3) select a suitable deposition temperature or annealing temperature, with a compromise between thermal stress and intrinsic stress; (4) use some interlayers to reduce large tensile stress in some TiNi films; (5) perform post-annealing, ion beam post-modification, or in-situ ion beam modification during sputtering in order to reduce intrinsic stress, (6) select suitable substrate to reduce thermal stress.

### D. Frequency response

One of the challenges for the successful application of TiNi films is effective reduction of hysteresis and increase in operating frequency. External heat generation and dissipation are necessary in generating phase transformation and actuation, and the response speed of NiTi microactuators is mainly limited by their cooling capacities. The binary Ti-Ni alloy films have a large temperature hysteresis of about 30°C. The hysteresis could be slightly reduced by decreasing the cyclic temperature amplitude and/or increasing working stress. R-phase transformation usually has a very small temperature hysteresis, which is useful for MEMS applications. However, the problem is that the strain and stress (or force) generated are too small to be of many practical uses. Addition of Cu in TiNi films is effective in reducing the hysteresis [15]. Compared with TiNi binary alloy, TiNiCu alloys also show less composition sensitivity in transformation temperatures, lower martensitic yield stress, and superior fatigue property, etc., which makes them more suitable for microactuator application. However, the transformation temperatures of TiNiCu films decrease slightly, and the transformation becomes weaker with the increase of Cu contents. Also the film becomes brittle when Cu content is higher than 10 at%.

### E. Adhesion and interfacial analysis

When TiNi films are deposited on Si substrate, there exist interfacial diffusion and chemical interactions at the interface whereby titanium and nickel silicides may form during high temperature deposition or post-deposition annealing. These interfacial reaction products could be complex, heterogeneous and metastable. Since the TiNi film thickness required in MEMS applications is usually less than a few microns, a relatively thin reaction layer could have significant adverse effect on adhesion and shape memory properties. In MEMS processes, there is a need for an electrically and thermally insulating or sacrificial layer. Thermally grown SiO<sub>2</sub> is often used as this sacrificial layer.

However, the adhesion of TiNi film on SiO<sub>2</sub> layer (or on glass and polymer substrate) is poor owing to the formation of a thin intermixing layer and the formation of a fragile and brittle TiO<sub>2</sub> layer (see Fig. 4) [16]. Adhesion of TiNi film on other substrates (such as Si<sub>3</sub>N<sub>4</sub>, polysilicon, etc.) is important for its successful MEMS applications, but few studies have been done so far.

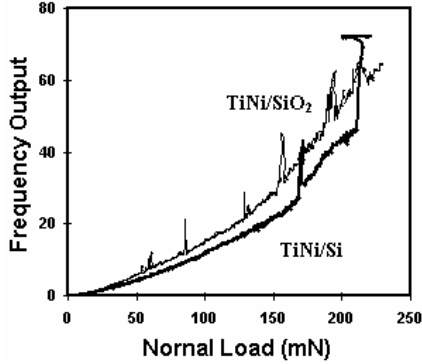


Fig. 4. Scratch results showing the adhesion failure of TiNi films deposited on Si and SiO<sub>2</sub> surface [16]

#### F. Performance degradation and fatigue consideration

Stability and fatigue have always been concerns in development of TiNi thin films for applications. Fatigue of TiNi films is referred to the non-durability and deterioration of the shape memory effect after millions of cycles. The repeated phase changes will alter the microstructure and hysteresis of the transformation and in turn will lead to changes in transformation temperatures, transformation stresses and strains. The performance degradation and fatigue of thin films are influenced by a complex combination of internal (alloy composition, lattice structure, precipitation, defects, film/substrate interface) and external parameters (thermo-mechanical treatment, applied maximum stress, stress and strain rate, the amplitude of temperature cycling frequency) after long term thermal-mechanical cycles. For freestanding films, there are some studies using tensile tests to characterize the fatigue problems. Results indicated that there need hundreds of cycles before the stability of shape memory effects. Ref. [17] studied the fatigue of the constrained TiNi films using the changes of recovery stress during cycling, and showed that the recovery stress of TiNi films from curvature measurement decreased dramatically in the first tens of cycles, and becomes stable after thousands of cycles (with one example shown in Fig. 5). Transformation temperatures also changed dramatically during cycling. This reduction of the recovery stress is believed to result from the dislocation movement, grain boundary sliding, void formation, or partial de-bonding at the film/substrate interfaces, non-recoverable plastic deformation, changes in stress, etc [4]. The repeated phase changes will alter the microstructure and hysteresis of the transformation and in turn lead to changes in transformation temperatures, stresses and strains.

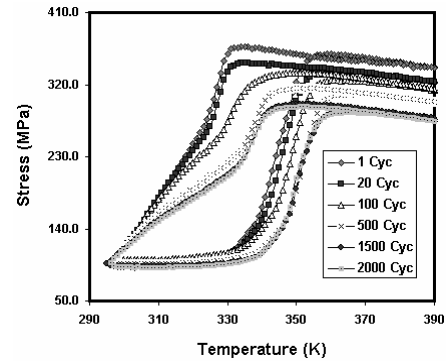


Fig. 5. Hysteresis evolution of Ti52.5Ni film on Si substrate after thermal cycling in different cycles and become stable after 2000 cycles

#### G. Functionally graded and composite TiNi based films

To further improve the properties of TiNi films, multi-layer, composite or functionally graded TiNi based films can be designed. So far, there are different design models for the functionally graded TiNi thin films. The first type is through the gradual change in composition (Ti/Ni ratio), crystalline structures, transformation temperatures, and/or residual stress through film thickness. As the Ti or Ni content changes in the micron-thick film, the material properties could change from pseudo-elastic (similar to rubber) to shape memory. The seamless integration of pseudo-elastic with shape memory characteristics produces a two-way reversible actuation, because residual stress variations in thickness will enable biasing force to be built inside the thin film. These functionally graded TiNi films can be easily prepared by sputtering deposition with slightly changing the target powers during deposition. Another novel way is to vary the target temperature during sputtering, and the films produced by hot targets have compositions similar to that of the target while films produced from cold target are Ti deficient.

The second type of functionally graded films involves new materials and functions other than TiNi films. Recently we [18,19] explored the deposition of a functionally graded TiN/TiNi layer to fulfill this purpose. The presence of an adherent and hard TiN layer (300 nm) on TiNi film (3.5  $\mu$ m) formed a good passivation layer (to eliminate the potential Ni release), and improved the overall hardness, load bearing capacity and tribological properties without sacrificing the shape memory effect of the TiNi film. Some surface modification methods, such as irradiation of TiNi films by electrons, ions (Ar, N, He, Ni or O ions), laser beams, neutrals can be used (1) to modify the surface physical, mechanical, metallurgical, wear, corrosion and biological properties for application in hostile and wear environment; (2) to cause lattice damage and/or alter the phase transformation behaviors along thickness of film, forming novel two-way shape memory actuation.

Other functionally graded or composite designs include the combination of TiNi films with piezoelectric, ferromagnetic, or magnetostrictive thin films [20]. Response time of the piezoelectricity mechanisms (PZT films) is fast, but the displacement is relatively small. TiNi film, on the

other hand, has a large force-displacement, but with slow response frequency. By coupling TiNi and PZT films to fabricate a new hybrid heterostructure composite or functionally graded films, it is possible to tune or tailor the static and dynamic properties of TiNi thin films, which may generate a larger displacement than conventional piezoelectric or magnetostrictive thin films and have an improved dynamic response compared with that of single layer TiNi films. However, the complexity of the fabrication processing, the interfacial diffusion and adhesion, and dynamic coupling of dissimilar components remain tough issues for these types of composite thin films.

#### IV. MEMS APPLICATIONS OF TINI FILMS

The main advantages of TiNi thin film include high power density, large displacement and actuation force, low operation voltage, etc. The main problems include: (1) Low energy efficiency, low dynamic response speed and large hysteresis. (2) Non-linearity and complex thermo-mechanical behavior and ineffectiveness for precise and complex motion control and force tracking; (3) High cost of TiNi films and difficulty in control of composition and mechanical properties; (4) Potential degradation and fatigue problems. Even with the above disadvantages, TiNi thin film is still considered as a core technology for actuation of some MEMS devices, where large force and stroke are essential and in conditions of low duty cycles or intermittent operation, and in extreme environment, such as radioactive, space, biological and corrosive conditions.

Freestanding films usually show intrinsic “two-way” shape memory effect, with large displacement, but relatively small little force in actuation. This is applicable in microsensors, microswitches or micropositioners. The nature of this “two-way” shape memory effect could be due to: (1) intrinsic residual stress in TiNi films; (2) compositional gradient through film thickness; (3) existence of R-phase; (4) hot shaping and aging of TiNi film resulting in  $Ti_3Ni_4$  precipitates. The constrained film/substrate actuators could provide large actuation force, but sacrifice the deflection (or strain). The substrate may act as an effective biasing force, thus creating a mechanical “two-way” shape memory effect.

Since TiNi films can provide large forces for actuation and large displacement, therefore, most applications of TiNi films in MEMS are focused on microactuators, such as micropumps, microvalves, micro-grippers, springs, microspacers, micro-positioners, and microrappers, etc. Fig. 6 shows one examples of microcantilever with TiNi microelectrode. TiNi thin films are sensitive to environmental changes such as thermal, stress, magnetic or electrical fields thus should be ideal for applications in microsensors, for example, optical switch, micromirror, etc. Fig. 7 and 8 shows two examples of micromirror structures. However, only a few studies and applications are reported in this respect.

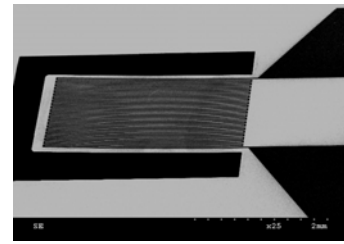
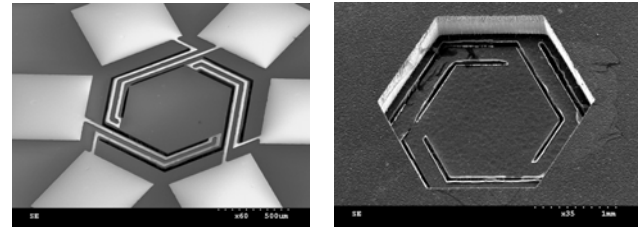


Fig. 6. A Si microcantilever structure with TiNi electrodes



(a)

(b)

Fig.7. TiNi micromirror structure with a Si cap acts as top mirror and the arms fabricated with TiNi/Si beam structure (a) top view; (b) bottom view

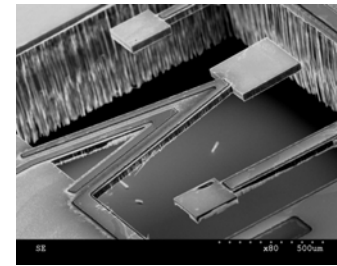


Fig. 8. A micromirror structure actuated by TiNi electrode

High passive and active damping capacity is considered as one of the important functional properties of shape memory alloys. Since the process involves the hysteresis movement of interfaces (martensite variant interfaces, like twin boundaries, phase boundaries, lattice defects), thus a large amount of energy is dissipated upon cycling. The changes of damping capacity of TiNi films have been used to study their phase transformation behaviors. Damping capacity is temperature and frequency dependent and peaks in the vicinity of the martensitic transformation temperature, thus these factors can be explored for the design of anti-vibration damping structures. In hard disk drives, the positioning accuracy of the read/write heads strongly depends on the inherent dynamic characteristics of the head actuator assembly (i.e., vibrations of the head actuator assembly system). TiNi film with large damping property is promising in minimizing the vibrations during the operations of the hard disk drive. However, fabrication of the actual damping devices must take into account factors of economics, reliability, versatility and construction needs. Also important to be considered are film stress and potential distortion of structures.

Good wear resistance is an important property required of some MEMS and biomedical applications, such as pumps, grippers, valves, etc. It was reported that bulk TiNi alloys in austenite exhibit good wear resistance (due to its rapid work hardening and pseudoelastic properties). However, poor wear

resistance of B19' (martensite) and high coefficient of friction are potential problems. In the case of shape memory alloy films, interfacial adhesion, large coefficient of friction and potential stress are other major concerns for their tribological application. Functionally graded layer design (TiN/TiNi) or surface modifications as discussed in section 5.1 could provide viable solution.

Bulk TiNi is a common and well-known material for the medical industry. At present, increasing attention has also been paid to use TiNi thin film into minimally invasive surgery, microstents and bioMEMS applications. Some microactuators made from TiNi thin films may be used to infuse drugs, or placed in strategic locations in the body to assist circulation. Glass, silicon and polymers are the mostly common used substrates for biological applications. However, high deposition or annealing temperatures for preparation of TiNi films and the poor adhesion on these substrates pose the potential problems. Superelasticity of TiNi, a non-linear pseudoelasticity as much as 7-10% strain, has already found many applications for bulk materials, but few explorations are carried out in MEMS applications so far using thin films. TiNi thin film SMA in its super-elastic state is promising for some compliant elements in MEMS devices.

Since thin films with nanometer grains (about tens or hundreds of nanometers) still show shape memory effect, it is promising to fabricate nano-scale SMA thin film structures with the aid of precision tools (such as focused ion milling or FIB). These structures may be able to perform physical actuation (push, pull, etc) at nano-scale. Possible difficulties of TiNi films in nanoscale structures may include: (1) large amount of oxygen and carbon adsorption on TiNi surface due to the extremely reactive nature of Ti elements, and the oxygen diffusion depth could be as large as tens of nanometers; (2) the difficulty in fabrication and manipulation of these nanostructure, although laser beam, or FIB is promising.

## V. CONCLUSIONS

Development of TiNi based SMAs thin films and microactuators witnessed a considerable progress in recent years. Some important issues pertaining to the preparation of high performance shape memory TiNi films using sputtering methods and their MEMS applications were reviewed in this paper. Successful application of TiNi thin films in MEMS requires consideration of the following issues: preparation and characterization, residual stress and adhesion, frequency improvement, fatigue and stability, patterning and modeling of behavior. At microscale, TiNi actuators outperform other actuation mechanisms in work/volume (or power/weight) ratio, large deflection and force, but with a relatively low frequency (less than 100 Hz) and efficiency as well as non-linear behavior. More functional and complex designs based on TiNi film devices are needed with multiple degrees of freedom and compact structure. TiNi film based microactuators will find potential applications in medicine, aerospace, automotive, and consumer products. Miniature TiNi actuated devices based

on sputtered TiNi films are ready for the huge commercial market, especially for medical microdevices and implantable applications.

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